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Indian Standard

SPECIFICATION FOR ETHYL CHLORIDE, TECHNICAL

UDC 661·723·62

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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Indian Standard

SPECIFICATION FOR ETHYL CHLORIDE, TECHNICAL

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Indian Standard

SPECIFICATION FOR ETHYL CHLORIDE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 12 January 1973, after the draft finalized by the Organic Chemical (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Ethyl chloride is used in the manufacture of tetraethyl lead and as an alkylating agent. It is also employed as a refrigerant, solvent and anaesthetic.

0.3 Ethyl chloride vapours are highly flammable and form explosive mixture with air. Inhalation of high concentrations of ethyl chloride can cause anaesthetic effects and continued contact with rapid evaporation from the skin can produce frostbite. Accumulation of its vapours in closed, unventilated areas where open flames, electric sparks, exposed electrical equipment or static electricity are present, can cause explosion. Its vapours, being 2·2 times heavier than air, are likely to settle to the ground level if adequate natural or forced ventilation is absent.

0.4 This standard contains clause **2.4** which calls for agreement between the purchaser and the supplier.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for ethyl chloride, technical for industrial use.

2. REQUIREMENTS

2.1 Description — The material is gaseous at ordinary temperatures and pressures, but usually compressed to a colourless, mobile, inflammable and very volatile liquid. It has pleasant and ethereal odour and burning taste.

*Rules for rounding off numerical values (*revised*).

2.2 Solubility—The material shall be completely soluble in rectified spirit (conforming to IS : 323-1969*) and ether, solvent grade (conforming to IS : 336-1964†) in all proportion.

2.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A are given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR ETHYL CHLORIDE, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Relative density* at 0°C/15°C	0·921 to 0·926	A-2
ii)	Boiling point range	12·0°C to 12·8°C	A-3
iii)	Residue on evaporation, percent by mass, *Max*	0·005	A-4
iv)	Moisture	To pass the test	A-5
v)	Acidity (as HCl), percent by mass, *Max*	0·005	A-6
vi)	Ionizable chlorides	To pass the test	A-7
vii)	Ethyl alcohol content	To pass the test	A-8
viii)	Ethyl chloride content, percent by mass, *Min*	99·0	A-9

*Relative density is the term adopted for specific gravity with water as reference substance by the International Organization for Standardization (ISO).

2.4 Optional Requirement—Subject to agreement between the purchaser and the supplier, the material may also satisfy the following requirements when tested according to the method prescribed in Appendix B:

a) Vinyl chloride, percent by mass, *Max* 0·10

b) 1·1 Dichloroethane, percent by mass, *Max* 0·10

c) 1·2 Dichloroethane, percent by mass, *Max* 0·05

3. PRECAUTIONS IN STORING AND HANDLING

3.1 Ethyl chloride is a highly flammable, volatile substance and therefore smoking and carrying of matches in locations where ethyl chloride is stored, handled or used shall be prohibited. No open flames or spark producing devices of any kind shall be permitted in or around locations, buildings or equipment where ethyl chloride is stored, handled or used.

*Specification for rectified spirit (*revised*).

†Specification for ether (*revised*).

Only non-sparking tools should be used and even then care should be exercised that heavy blows are not struck where ethyl chloride vapour may be present (*see also* **0.3**).

4. PACKING AND MARKING

4.1 Packing — The material shall be supplied in suitable steel cylinders.

4.1.1 The cylinder shall comply with the requirements for cylinders for liquified gases given in the Gas Cylinder Rules, 1940 of the Government of India, with such modifications as may be ordered from time to time by the Chief Inspector of Explosives, Government of India or any other duly constituted authority.

4.2 Marking — The containers shall be labelled with the following information:

a) Name of the material;

b) Manufacturers' name and his recognised trade-mark, if any;

c) Net mass of the contents;

d) Batch number and/or lot number, in code or otherwise; and

e) The following cautionary markings in clear bold letters:

ETHYL CHLORIDE

Danger ! Extremely flammable.

Keep away from heat, sparks and open flame.

Use with adequate ventilation.

Keep container closed, out of sun, and away from heat.

4.2.1 In addition to the above, the marking and labelling of cylinders shall be in accordance with the requirements for cylinders for liquefied gases given in the Gas Cylinder Rules, 1940 of the Government of India, with such modifications as may be ordered from time to time by the Chief Inspector of Explosives, Government of India or any other duly constituted authority.

4.2.2 The cylinders shall also be marked with the symbol for danger of ignition as specified in IS: 1260-1958*.

*Code of symbols for labelling of dangerous goods.

4.2.3 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix C.

APPENDIX A

(*Clause* 2.3)

METHOD OF TEST FOR ETHYL CHLORIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS:1070-1960*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF RELATIVE DENSITY

A-2.1 Procedure — Cool sufficient quantity of the material to about −5°C by surrounding the container with a mixture of methyl alcohol and solid carbon dioxide. Transfer the cooled material to a hydrometer cylinder standing in a bath of melting ice and insert the hydrometer (calibrated at 15°C) and a thermometer. Note the reading on the hydrometer when the temperature on the thermometer records 0°C and remains constant at that temperature during observation of the reading.

NOTE — The correct hydrometer reading is that point on the hydrometer scale at which the surface of the liquid cuts the scale. This point may be determined by placing the eye slightly below the level of the liquid and slowly raising it until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale.

*Specification for water, distilled quality (*revised*).

A-3. DETERMINATION OF BOILING RANGE

A-3.1 Apparatus

A-3.1.1 *Rubber Bung* — with two holes, one for the exit glass tube and the other for the thermometer.

A-3.1.2 *Measuring Cylinder* — 100 ml capacity (*see* IS : 878-1956*).

A-3.1.3 *Thermometer* — of 0°C to 15°C range or any other suitable range with 0·2°C graduation. The thermometer shall bear certificate of the National Physical Laboratory of India or any other institution authorised for the purpose.

A-3.2 Procedure — Fit to the rubber bung a short exit glass tube of internal diameter 6 mm. Insert the thermometer through the second hole of the bung. Cover the bulb of the thermometer with a piece of very fine muslin, free from sizing materials and grease, in such a manner that one end hangs 1 cm below the bulb. Dry the glass cylinder. Cool the glass cylinder and the material to be tested separately in ice-water and transfer 100 ml of the cooled material to the glass cylinder. Insert the bung and adjust the thermometer in such a manner that the hanging end of the muslin dips into the liquid and the bulb is above the surface. Replace the ice-water around the cylinder with water between 24 and 26°C and observe the temperature when 5 ml has evaporated and again when 5 ml remains. Lower the thermometer continuously to maintain its position relative to liquid surface throughout the test.

A-3.3 Correction of Thermometer Readings

A-3.3.1 *Error of Scale* — In all thermometer readings, make the corrections as indicated on the certificate of the instrument.

A-3.3.2 *Correction for Barometric Pressure* — If the barometric pressure prevailing during the determination is 760 mmHg, no correction need be applied to the specified temperature and the thermometer scale as corrected under **A-3.3.1** may be used as such. If the prevailing barometric pressure deviates from 760 mmHg, the specified temperature be corrected as follows:

a) For every 10 mmHg above 760 mmHg, add 0·35°C to the specified temperature; and

b) For every 10 mmHg below 760 mmHg, substract 0·35°C from specified temperature.

NOTE — These corrections are valid only for pressure above 700 mmHg.

*Specification for graduated measuring cylinders.

A-4. RESIDUE ON EVAPORATION

A-4.1 Apparatus

A-4.1.1 Porcelain Basin — 150 ml capacity (conforming to IS:2837-1964*).

A-4.1.2 *Oven* — capable of maintaining temperature of 105 ± 2°C.

A-4.2 Procedure — Weigh the porcelain basin. Cool the material to 0°C and by means of a pipette, with rubber bulb aspirator, transfer 100 ml of the material to the tared porcelain basin. Allow the material to evaporate at room temperature and then heat the residue in the oven at 105 ± 2°C for 30 minutes. Cool in a desiccator and weigh accurately.

A-4.3 Calculation

$$\text{Residue on evaporation, percent by mass} = \frac{M_1 - M}{V \times d} \times 100$$

where

M_1 = mass in g of the basin with the residue,

M = mass in g of the basin,

V = volume in ml of the material taken for the test, and

d = relative density of the material as determined under **A-2.**

A-5. TEST FOR MOISTURE CONTENT

A-5.1 Apparatus

A-5.1.1 *Boiling Flask* — narrow-necked, conical and of capacity 50 ml (*see* IS:1381-1959†).

A-5.2 Reagent

A-5.2.1 *Copper Sulphate* (*Blue Vitriol*) — exsicatted.

A-5.3 Procedure — Dry the boiling flask. Introduce into it by means of a pipette 25 ml of the material maintained at 0°C and 0·5 to 1·0 g of the exsicatted copper sulphate and observe for any colour change.

A-5.3.1 The material shall be regarded to have passed the test if the colour of the copper sulphate does not change.

A-6. DETERMINATION OF ACIDITY

A-6.1 Apparatus

A-6.1.1 *Separating Funnels* — 200 ml capacity (*see* IS:1575-1960‡).

*Specification for porcelain crucibles and basins.
†Specification for boiling flasks (narrow-necked).
‡Specification for separating funnels.

A-6.1.2 *Boiling Flask* — narrow-necked, conical and of 100 ml capacity (*see* IS:1381-1959*).

A-6.2 Reagents

A-6.2.1 *Standard Sodium Hydroxide Solution* — 0·01 N.

A-6.2.2 *Phenolphthalein Indicator* — Dissolve 0·5 g of phenolphthalein in 100 ml of rectified spirit (*see* IS:323-1959†). Add standard sodium hydroxide solution until the indicator is faint pink.

A-6.3 Procedure — Cool the material to 0°C and then introduce by means of a pipette 25 ml of the cooled material into the separating funnel. Add 25 ml of distilled water cooled to 5°C. Hermetically close the funnel by means of a glass or rubber stopper, carefully shake the liquid and then allow the layers to separate. Repeat this thrice. After separation of the layers, remove the water layer and collect it in the boiling flask. Add 2 to 3 drops of phenolphthalein indicator and then titrate with the standard sodium hydroxide solution to the end point.

A-6.4 Calculation

$$\text{Acidity (as HCl), percent by mass} = \frac{0{\cdot}14586\ VN}{D}$$

where

V = volume in ml of the standard sodium hydroxide solution used,

N = normality of standard sodium hydroxide solution, and

D = relative density of the materials (*see* **A-2**).

A-7. TEST FOR IONIZABLE CHLORIDES

A-7.1 Preparation of Test Sample — Cool the material to 0°C, pipette out 20 ml of the cooled material into a 100 ml boiling flask, add 20 ml of water at 0°C and shake. Then allow the material to evaporate. Reserve the residual liquids for tests under **A-7.3** and **A-8.2**.

A-7.2 Reagent

A-7.2.1 *Silver Nitrate Solution* — Dissolve 4·25 g of silver nitrate in 100 ml of water.

A-7.3 Procedure — Take 5 ml of the residual liquid (*see* **A-7.1**) in a test-tube and add 1 ml of silver nitrate solution. Observe if any turbidity is produced.

*Specification for boiling flasks (narrow-necked).
†Specification for rectified spirit (*revised*).

A-7.3.1 The material shall be regarded to have passed the test if no turbidity is produced.

A-8. TEST FOR ETHANOL CONTENT

A-8.1 Reagents

A-8.1.1 *Potassium Dichromate Solution* — approximately 2 N.

A-8.1.2 *Sulphuric Acid* — analytical reagent grade (*see* IS : 266-1961*).

A-8.2 Procedure — Take 5 ml of the residual liquid (*see* **A-7.1**) in a test-tube, add 2 drops of potassium dichromate solution and 2 ml of sulphuric acid, and warm. Note the colour and the smell produced.

A-8.2.1 The material shall be regarded to have passed the test if no odour of acetaldehyde is produced and the solution do not show any greenish colour.

A-9. DETERMINATION OF ETHYL CHLORIDE CONTENT

A-9.0 Outline of the Method — The material is hydrolysed with a known amount of standard alcoholic potassium hydroxide solution. The exact amount of standard alcoholic potassium hydroxide solution required for the hydrolysis is determined and from that ethyl chloride content is calculated.

A-9.1 Reagents

A-9.1.1 *Alcoholic Potassium Hydroxide Solution* — 2 N.

A-9.1.2 *Phenolphthalein Indicator* — same as in **A-6.2.2.**

A-9.1.3 *Standard Hydrochloric Acid* — 2 N.

A-9.2 Procedure — Introduce quickly about 1·5 g of the material into a tared glass ampoule provided with a long neck, containing 25 ml of alcoholic potassium hydroxide solution. Surround the ampoule with freezing mixture and seal the neck hermitically. Weigh accurately after bringing the ampoule to room temperature, and heat in a water-bath for 2 hours. Cool and carefully break the neck of the ampoule and dilute the contents to 100 ml and titrate with standard hydrochloric acid using phenolphthalein indicator to the end point.

A-9.3 Calculation

$$\text{Ethyl chloride content, percent by mass} = \frac{6{\cdot}452\ (V_1N_1 - V_2N_2)}{M_1 - M_2}$$

where

V_1 = volume in ml of alcoholic potassium hydroxide solution taken in the ampoule,

*Specification for sulphuric acid (*revised*).

N_1 = normality of the standard potassium hydroxide solution,

V_2 = volume in ml of standard hydrochloric acid,

N_2 = normality of standard hydrochloric acid,

M_1 = mass in g of the glass ampoule containing standard alcoholic potassium hydroxide solution and the material under test, and

M_2 = mass in g of the glass ampoule containing standard alcoholic potassium hydroxide solution.

APPENDIX B

(*Clause* 2.4)

DETERMINATION OF VINYL CHLORIDE, 1:1 DICHLOROETHANE AND 1:2 DICHLOROETHANE

B-0. GENERAL

B-0.1 Outline of the Method — The chlorinated compounds like vinyl chloride, 1:1 dichloroethane and 1:2 dichloroethane present in ethyl chloride in small amounts are determined by gas chromatography.

B-1. APPARATUS

B-1.1 Gas Chromatograph — Any commercially available gas chromatograph with a thermal conductivity detector may be used. The operating conditions of one of the suitable columns are given below:

Column	
Material	Stainless steel
Length	3 m
Internal diameter	4 mm, approximately
External diameter	6 mm, approximately
Support	Chromosorb P (30 to 60 mesh)
Stationary phase	Diethyl sebacate
Preparation of the stationary phase and support	25 percent by mass of diethyl sebacate on the chromosorb P (30 to 60 mesh)

B-1.2 Detector — Thermal conductivity cell type of detector.

B-1.3 Recorder — Full scale deflection: 1 second.

B-2. TEST SUBSTANCES

B-2.1 The following components are estimated:

a) Vinyl chloride,

b) Ethyl chloride,

c) 1 : 1 Dichloroethane, and

d) 1 : 2 Dichloroethane.

B-3. PROCEDURE

B-3.1 Operating parameters of gas chromatograph with the above column (*see* **B-1.1**) are as follows :

a) Injection port	Temperature 130°C
b) Column temperature	85 ± 1°C
c) Carrier gas	Hydrogen (99·99 percent purity minimum on *v/v* basis). Flow rate: 80 ml/minute. Inlet pressure: approximately 0·6 atm
d) Detector	Temperature 85 ± 1°C
e) Bridge current	150 mA
f) Recorder	Chart speed: 1·25 cm/min

B-3.2 Calibration — Internal normalization is the method used for calibration of the results of the analysis. Because of low contents of impurities it is not necessary to use correction factors for converting the percentage of the areas into percentage by mass, so that these factors in the calculation formula have the value $K = 1$. In case of samples with higher contents of impurities, there is a necessity for determing correction factors; this should be done by using a known volume of cooled ethyl chlorides with known amount of vinyl chloride, 1 : 1 dichloroethane and 1 : 2 dichloroethane. The mixture is run through the chromatograph. This would give the positions at which each components would elute. By comparison of the actual values obtained for the standard and the composition of the standard prepared, the correction factor for the component may be calculated.

B-3.3 Tests — Cool the sample in a test-tube using ice and salt mixture, along with a microlitre syringe. After it is cooled sufficiently, draw the sample into the syringe. Introduce about 6 to 7 microlitres of the material into the chromatograph. Wait for the optimum conditions for the attenuation which is approximately 30 minutes.

B-3.4 Interpretation of Chromatogram — Elution order of the components is vinyl chloride, ethyl chloride, 1 : 1 dichloroethane and 1 : 2 dichloroethane. Identification is done with the aid of a test mixture.

B-3.5 Calculation

$$X_i = \frac{100 \times K_i \times A_i}{\Sigma K_i \cdot A_i}$$

where

X_i = percentage by mass of component i,

K_i = correction factor of component i, and

A_i = peak area due to component i [peak height (×) width at half height].

APPENDIX C

(*Clause* 5.1)

SAMPLING OF ETHYL CHLORIDE, TECHNICAL

C-1. GENERAL PRECAUTIONS

C-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

C-1.1 The sampling instrument shall be clean and dry where used.

C-1.2 Precautions shall be taken while drawing samples (*see* **0.3** and **3**).

C-1.3 The samples shall be placed in a suitable, clean, dry and air-tight glass containers.

C-1.4 The sample containers shall be of such a size that they are almost but not completely filled by the sample.

C-1.5 Each sample container shall be sealed air-tight with a glass stopper after filling and marked with full details of sampling, the name of manufacturer and other important particulars of consignment.

C-2. SAMPLING APPARATUS

C-2.1 A copper coil with threaded nipple at one end shall be used for sampling (*see* Fig. 1). The threaded nipple is connected to the lower valve of the cylinder and kept in a horizontal position. The coil is kept immersed in an ice and salt-bath. On opening the lower valve slowly the sample collects in the bottle.

C-3. SCALE OF SAMPLING

C-3.1 Lot—All the containers in the single consignment of material drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

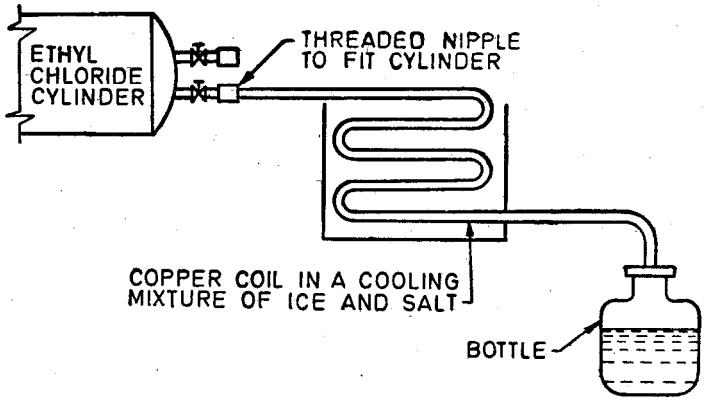

FIG. 1 SAMPLING APPARATUS

C-3.1.1 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of this specification.

C-3.2 The number (n) of containers to be selected for the test shall depend on the size (N) of the lot and shall be in accordance with Table 2.

C-3.3 These containers shall be selected at random from the lot. To ensure randomness of selection a random number table, as agreed to between the purchaser and the supplier shall be used (*see* IS : 4905-1968*). In case such a table is not available, the following procedure shall be adopted:

Starting from any container, in the lot, count them as 1, 2, 3,..., up to r and so on in one order, where r is the integral part of the value N/n [N being the total number of containers in the lot and n the number of containers to be chosen (*see* Table 2)]. Every rth containers thus counted shall be separated until the requisite number of containers is obtained from the lot.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING
(*Clauses* C-3.2 *and* C-3.3)

LOT SIZE	NO. OF CONTAINERS TO BE SELECTED
N	n
(1)	(2)
Up to 15	3
16 to 40	4
41 ,, 65	5
66 ,, 110	7
111 and above	10

NOTE — When the size of the lot is 3 or less, all the containers shall be sampled.

*Methods for random sampling.

C-4. TEST SAMPLES AND REFEREE SAMPLES

C-4.1 Cool the sampling apparatus and the sample containers in a freezing bath at a temperature not higher than −5°C. From each selected container withdraw about 1 500 ml of the material by passing through a sampling apparatus (*see* Fig. 1). Immediately transfer approximately equal volume of the material into three sample containers of capacity not less than 500 ml. Each selected container will have three samples containers. The sample containers shall be properly sealed with stoppers and labelled with the particulars as given in **C-1.5**. The material in each sample container shall constitute an individual test sample. The sample containers shall be placed in the freezing bath till they are ready for testing. These individual test samples shall be separated into three identical sets of test samples in such a way that each set has a sample representing each container. One of these three sets shall be marked for the purchaser, another for the supplier and the third for the referee.

C-4.2 If it is necessary to transport the sample, transfer to a cylinder which shall comply with the requirements for cylinders for liquefied gases, given in the Gas Cylinder Rules, 1940 of the Government of India, with such modification as may be ordered from time to time by the Chief Inspector of Explosives, Government of India or any other duly constituted authority.

C-4.3 Referee Samples — Referee samples shall consist of a set of test samples (**C-4.1**) marked for this purpose and shall bear the seales of the purchaser and the supplier. These shall be kept at a place agreed to between the two.

C-5. NUMBER OF TESTS

C-5.1 All the individual test samples from each lot prepared as in **C-4.1** shall be tested separately for all the requirements as given in Table 1. In the contigencies, such as loss, spoilage and spilling of the purchasers' test samples or if the supplier so desires, the test samples marked for the supplier may be tested. In case of dispute, the referee test samples shall be tested and the test result obtained on the referee samples shall be considered as final.

C-6. CRITERIA FOR CONFORMITY

C-6.1 A lot shall be declared as conforming to the requirements of this specification if all the individual test results satisfy the relevant requirements given in Table 1.

INDIAN STANDARDS
ON
ORGANIC CHEMICALS (MISCELLANEOUS) MATERIALS

IS:

245-1970 Trichloroethylene, technical (*second revision*)

501-1963 Oxalic acid, technical and analytical reagent (*revised*)

716-1970 Pentachlorophenol (*first revision*)

717-1969 Carbon disulphide, technical (*first revision*)

718-1970 Carbon tetrachloride (*first revision*)

869-1969 Ethylene dichloride (*first revision*)

880-1956 Tartaric acid

3321-1965 Formaldehyde solution

4105-1967 Styrene (vinyl benezene)

4306-1967 Hexamethylenetetramine (hexamine)

4566-1968 Methylene chloride (dichloromethane), technical

5149-1969 Maleic anhydride, technical

5158-1969 Phthalic anhydride, technical

5254-1969 Acetanilide

5271-1969 Paraformaldehyde

5295-1969 Ethylene glycol

5296-1969 Chloroform, technical and analytical

5297-1969 Perchloroethylene (tetrachloroethylene), technical

5341-1969 Benzyl chloride, technical

5464-1970 Citric acid, monohydrate

5573-1969 Ethylene oxide

5591-1969 Chlorobenzene

5592-1969 Monochloroacetic acid

5992-1970 *p*-Dichlorobenzene, technical

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